

English translation of JP-B S31-007808

Method for continuously producing metal titanium

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a vertical sectional view illustrating one embodiment of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to a process for continuously producing titanium metal, comprising the steps of concurrently injecting a liquid of an alkaline metal or an alkaline earth metal or a mixture thereof and a liquid or vapor of titanium tetrachloride into a reaction chamber at normal temperatures or a relatively low temperature through a nozzle; allowing them to perform reduction reactions at relatively low temperature of about 700 to 900.° C, which is higher than the melting point of chloride of a reducing agent, only by their reaction heats; collecting a mixture of titanium and salts produced in the form of a slime, below a molten salt; and heating or cooling a tapping device to transfer the mixture to a vacuum furnace so that the mixture is subjected to vacuum distillation.

In the present invention, the reactions of reduction are carried out by injecting, as a nebulosus or gaseous state, a liquid of an alkaline metal or an alkaline earth metal or a mixture thereof simultaneously with a liquid or vapor of titanium tetrachloride mixed with or without argon into a vapor zone of inactive gas such as argon in the reaction chamber through a nozzle composed of coaxial double tubes. In that case, since the reducing agents for metals have melting points listed below, they can be introduced into the reaction chamber at a temperature ranging from normal temperature to 600 °C.

Melting point

| | Mg | Ca | Na | K |
|-----------|-----|-----|------|------|
| Metal mp. | 650 | 850 | 97.5 | 63.5 |

Alloy Na·K alloy

(Range of alloys being a liquid at a temperature of 50° C and below)

Na 3~75 %

Ca·Mg alloy

(Range of alloys being a liquid at a temperature of 500° C and below)

Ca 75~82 %

(Range of alloys being a liquid at a temperature of 600° C and below)

Ca 8~23 %

Ca 70~85 %

Note: Sodium produced by direct electrolysis of sodium chloride contains approximately 1 % of Ca, and its melting point is about 150 ° C.

Further, the reduction reactions may be initiated by introducing an alloy (into the chamber) to cause them self-activation, said alloy containing a large quantity of the alkaline metal or alkaline earth metal and being heated to a temperature of 500 °C and above. Then, the temperature in the chamber is maintained by their reaction heats to a temperature of about 700 to 900 °C, which is higher than the melting point of the chloride of reducing agent. Then, titanium metal and molten salt or mixed salts produced by the above reactions are allowed to fall in a collecting portion kept at about 700 to 800 °C, and the mixtures of titanium metal and the salts are allowed to respectively precipitate to the bottom of the molten salt due to difference in specific gravity. The molten salt thus precipitated in the collecting portion and titanium metal precipitated below the molten salt in the slime form are taken out through respective outlets.

In that case, titanium metal is produced by introducing the titanium metal into a vacuum furnace by heating a tapping device, heating the vacuum furnace to 1200 to 1300 °C to remove the salt and nonreacted metal by vaporization, and then collecting powdered titanium metal only after its agglutination. By alternately replacing the vacuum furnace with another one, it is possible to continuously produce titanium metal without interruption of reduction reactions.

According to the present invention, therefore, it is possible to supply reactants of the metal-reducing agent and titanium tetrachloride to a reaction chamber at extremely low temperatures when carrying out reactions for production of titanium metal. The initiation of reactions is carried out by self-ignition caused by injection of molten metal. Also, it is sufficient to use the reaction heats as a heat source required for reactions. In addition, the temperature control can be done freely by controlling the injection quantity of the reaction materials. Also, since the produced titanium metal is mixed with the molten salt and stored in the form of slime below the molten salt, it is easy to handle the products as well as to prevent foreign matters of internal wall of the reaction chamber from getting into the reaction products.

Thus, the present invention makes it possible to easily and economically achieve continuous production of titanium metal with high purity.

The method of the present invention will be explained below with reference to the accompanying drawing.

Firstly, one embodiment of the titanium metal reduction furnace used in the process of the present invention will be outlined below. A furnace wall 1 is made of a heat-resisting metal or alloy such as stainless steel, titanium or titanium alloy. The furnace includes a reaction chamber 2 located at an upper portion, an enlarged furnace bosh 3 located at a middle portion, and a furnace bottom portion 4 kept its temperature constant to keep salts of reaction products in a molten state and tapered toward the bottom to collect reaction products therein. The furnace is provided at its top with a coaxial double tubular nozzle 5, 5' for concurrently injecting a liquid (or vapor) of titanium tetrachloride and a liquid of the reducing agent into the chamber from the top of the furnace. The furnace is also provided at the furnace bosh 3 with a pipe 6 for control of the internal pressure of the furnace and for replacement of atmospheres in the vapor zone.

The furnace is provided at its lower portion with a tapping hole 7 for taking out the molten salts, and a tapping device 8 for taking out the slurry of precipitated spongy titanium. The tapping device 8 allows the precipitate to start or stop the flow entering into a vacuum heating furnace 9 by heating or cooling a conduit connected thereto.

The furnace is wholly housed in a steel shell 10 serving as a supporting device and its internal gas pressure is equilibrated with that of the shell 10 to allow the furnace material to hold oxidation resistance and pressure resistance.

The nozzle for injecting titanium tetrachloride and a sodium-potassium alloy (Na 50%) is provided on the top of the furnace body like a bomb with an internal volume of 50 liters, the atmosphere in the furnace is replaced with argon gas through the pipe 6. Then, the reduction reaction is carried out by injecting titanium tetrachloride first by spraying and then injecting the molten sodium-potassium alloy heated to 500 ° C to ignite the furnace by itself, and controlling the injection quantities so as to keep the internal temperature of the furnace to the vicinity of 900 ° C.

By continuing the reduction reaction, a mixture of molten salts and powdered titanium in the form of slime is collected below the molten salt in the bottom portion of the furnace, and introduced into the underneath vacuum heating furnace through the tapping conduit as it is. Then, the inflow of the reaction products is stopped by cooling the tapping conduit with water to solidify the salt flowing through the conduit. In the vacuum furnace, the reaction products are heated to 1200 to 1300 ° C to evaporate the sodium chloride and potassium chloride remained therein as the molten salts, which are then introduced into a condensing device through a pipe 11. Simultaneously therewith, sponge titanium is agglutinated and stabilized. Then, titanium metal is taken out by cooling the vacuum furnace. Use of 800 g of titanium chloride and 530 g of sodium-potassium alloy yielded 172 g of sponge titanium. By using plural vacuum furnaces and replacing the vacuum furnace with another one, the reaction is carried out continuously.

Claim:

A process for continuously producing titanium by introducing titanium tetrachloride and a reducing agent into an inactive gas zone in a reaction chamber to react them with each other so as to produce titanium and a salt, said process comprising the steps of concurrently injecting an alkaline metal or an alkaline earth metal in a liquid phase or a mixture thereof, heated to a temperature of from a normal temperature to 700° C, and titanium tetrachloride in a liquid or vapor phase from a coaxial tubular nozzle into the reaction chamber to cause spontaneous ignition; maintaining the temperature in the reaction chamber at a temperature of substantially 700 to 900° C not lower than the melting point of the chloride of the reacting agent by using only the reaction heat of the reduction; collecting a mixture of the titanium and the salt produced in the form of slime in a lower side under the melted salt; and heating or cooling a tapping device to thereby transfer the mixture to a vacuum heating furnace so as to be subjected to vacuum distillation.

metal titanium -reducing furnace

